ALS IR News

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First Performance Results for the New Continuµm™ IR Microscope

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A second IR microscope and bench has been recently added to the ALS Beamline 1.4 complex. The FTIR bench is a Thermo Nicolet Nexus 870 which is capable of both rapid- and step-scan measurements. The IR microscope is a Thermo SpectraTech Contiuµm which has several new features not found on our Nic-Plan instrument. This new microscope has a dichroic element which allows visualization of the sample even while acquiring data. This is very convenient, especially with the synchrotron source as often the focused visible light spot from the synchrotron source can be observed indicating exactly where the beam is located on a sample. The optics in this microscope are infinity corrected which allows the easy addition of several types of optics which can assist in visualizing a sample, including visual and IR polarizers, Nomarski differential interference contrast (DIC) optics, and UV fluorescence with three difference filter cubes. All of these are available at ALS Beamline 1.4. This instrument is pictured in Figure 1, and was purchased through a DOE OBER grant to develop biological and biomedical applications of synchrotron-based infrared spectromicroscopy.

The new microscope has been temporarily installed at BL1.4.2 where we carried out initial performance tests comparing the internal IR source and the synchrotron beam as the source. Since signal to noise ration is a crucial parameter for good measurements, we measured a series of 100% reflection lines off of a gold-coated glass sample for both sources as a function of aperture size. We used an MCT-B detector, co-added 128 scans for background and sample measurements, at a spectral resolution of 4 cm⁻¹ and a scanning mirror velocity of 1.8988 cm/sec. Figure 2 shows the measured 100% lines for the EverGloTM thermal IR source and Figure 3 shows the results for the synchrotron source. Each measured line is annotated with the aperture size used and the resultant RMS noise value determined between 2450 and 2550 cm⁻¹. Since the focused size of the thermal IR source is greater than 70×70 microns, closing down the aperture size simply reduces the total IR signal

proportional to the area reduction. The noise level becomes significantly worse as the aperture is size is decreased, becoming essentially unusable at aperture sizes below 20×20 microns.

The focused spot size of the synchrotron source, however, is diffraction limited (3 to 10 microns in diameter) so its signal to noise ratio is only affected at aperture sizes smaller than 10 microns. The synchrotron is observed to have a better signal to

Figure 1. Photograph of the new Continu**m**n IR microscope (left) and Nexus 870 FTIR bench (right).



60

55

6000

5000

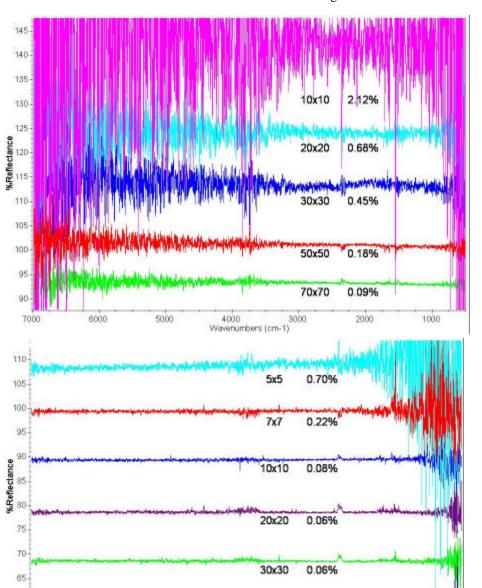


Figure 2. 100% reflectance lines showing the noise level of the EverGloTM thermal IR source when using smaller and smaller apertures. Noted beneath each curve are the aperture dimensions and the RMS noise determined between 2450 and 2550 cm⁻¹.

Figure 3. 100% reflectance lines showing the noise level of the ALS synchrotron IR source when smaller and smaller using Noted beneath each apertures. thecurve aperture dimensions and the RMS noise determined between 2450 and 2550 cm^{-1} . The diffractionlimited synchrotron spot size is not clipped until the aperture is below 10 microns, and even then it is primarily cutting off the longer wavelengths.

noise than the thermal IR source at all aperture sizes. The noise doesn't start to increase until the aperture size reaches 10×10 microns, and a usable signal is maintained even at the smallest aperture sizes available of 5×5 microns. Note that the longer wavelengths (lower wavenumbers) are being cutoff by the smallest aperture settings as is expected by the diffraction limited spot sizes (approximately the wavelength).

2000

1000

0.06%

70x70

4000

Wavenumbers (cm-1)

The signal to noise value at 2500 cm⁻¹ was obtained for each source and aperture setting measured by dividing the single beam intensity at this wavenumber value by the corresponding RMS noise value. The results are plotted in Figure 4. The signal to noise ratio for the thermal EverGloTM source drops rapidly as the aperture size decreases, whereas for the synchrotron source the signal to noise ratio remains essentially unchanged until the aperture size finally reaches the beam spot size of 10 microns. The synchrotron source's signal to noise ratio is more than 1000 times better than the thermal source for aperture sizes of 10 microns and smaller, which validates the calculated brightness advantage of a synchrotron IR beamline over a conventional thermal IR source for mid-infrared spectromicroscopy.

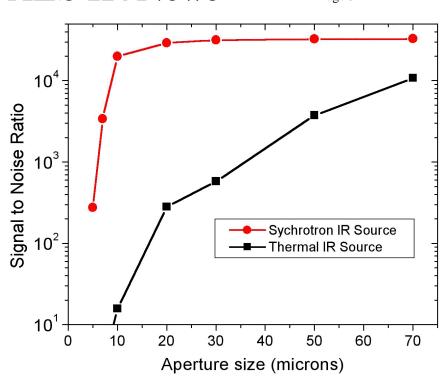


Figure 4. Comparison of the measured signal to noise ratio (on a logarithmic scale) for the synchrotron and thermal EverGloTM IR sources as a function of microscope aperture size. We observe a greater than 1000 times better signal to noise ratio for the synchrotron source compared to the thermal source for apertures 10 microns or smaller.

This Continuµm IR microscope and Nexus FTIR bench will later this year be placed permanently on ALS Beamline 1.4 with the synchrotron beam feeding both IR microscopes simultaneously without any loss of signal. This will be achieved by separately collimating the upstream and downstream ends of the bend magnet radiation. This will be part of a major rearrangement of the Beamline 1.4 complex toward the end of 2002 to make room for a new set of hard x-ray superbend beamline hutches from superbend 12.3. Once complete, these changes will double the amount of infrared microscopy beamtime that is available for IR users.

More detectors for this system are on order and will be installed before the fall. They will include a high sensitivity small element MCT-A* detector for the Continuµm, and a far-IR detector for the 870 bench. The Continuµm microscope will be optimized for biological and biomedical applications where the additional sample visualization techniques will be most advantageous. Further optimization of this new SR-IR spectromicroscopy endstation will continue as the equipment is moved into its permanent location on the ALS experimental floor.